

3-(2-Fluorophenylsulfinyl)-2,4,6-trimethyl-1-benzofuran

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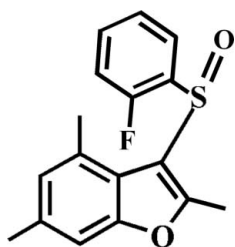
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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.043; wR factor = 0.114; data-to-parameter ratio = 17.0.

In the title compound, $\text{C}_{17}\text{H}_{15}\text{FO}_2\text{S}$, the 2-fluorophenyl ring makes a dihedral angle of $87.53(5)^\circ$ with the mean plane [r.m.s. deviation = $0.013(1)$ Å] of the benzofuran fragment. In the crystal, molecules are linked by weak $\text{C}-\text{H}\cdots\text{F}$, $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\pi$ interactions, forming a three-dimensional network.

Related literature

For background information and the crystal structures of related compounds, see: Choi *et al.* (2010); Seo *et al.* (2011).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{15}\text{FO}_2\text{S}$
 $M_r = 302.35$
 Monoclinic, $P2_1/c$
 $a = 13.6892(5)$ Å

$b = 6.0339(2)$ Å
 $c = 17.1786(7)$ Å
 $\beta = 92.741(2)^\circ$
 $V = 1417.32(9)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.24$ mm⁻¹

$T = 173$ K
 $0.32 \times 0.27 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.527$, $T_{\max} = 0.746$

13205 measured reflections
 3277 independent reflections
 2665 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.114$
 $S = 1.07$
 3277 reflections

193 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.36$ e Å⁻³
 $\Delta\rho_{\min} = -0.43$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 and Cg2 are the centroids of the C2–C7 and C12–C17 rings, respectively.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{O2}^{\text{i}}$	0.95	2.42	3.347 (2)	164
$\text{C10}-\text{H10C}\cdots\text{O2}^{\text{ii}}$	0.98	2.42	3.385 (2)	167
$\text{C11}-\text{H11A}\cdots\text{F1}^{\text{iii}}$	0.98	2.54	3.160 (2)	121
$\text{C11}-\text{H11B}\cdots\text{Cg1}^{\text{iv}}$	0.98	2.69	3.476 (2)	138
$\text{C15}-\text{H15}\cdots\text{Cg2}^{\text{v}}$	0.95	2.71	3.548 (2)	147

Symmetry codes: (i) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 2, -y, -z + 1$; (iv) $x, y - 1, z$; (v) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FY2099).

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supplementary materials

Acta Cryst. (2013). E69, o998 [doi:10.1107/S160053681301461X]

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Comment

As a part of our continuing study of 2,4,6-trimethyl-1-benzofuran derivatives containing 4-fluorophenylsulfinyl (Choi *et al.*, 2010) and 3-fluorophenylsulfinyl (Seo *et al.*, 2011) substituents in 3-position, we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the benzofuran unit is essentially planar, with a mean deviation of 0.013 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the 2-fluorophenyl ring and the mean plane of the benzofuran fragment is 87.53 (5)°. In the crystal packing, molecules are connected by weak C—H···F and C—H···O hydrogen bonds (Fig. 2 & Table 1), and by C—H··· π interactions (Fig. 3 & Table 1), forming a three-dimensional network.

Experimental

3-Chloroperoxybenzoic acid (77%, 291 mg, 1.3 mmol) was added in small portions to a stirred solution of 3-(2-fluorophenylsulfinyl)-2,4,6-trimethyl-1-benzofuran (343 mg, 1.2 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 4h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (hexane–ethyl acetate, 4:1 v/v) to afford the title compound as a colorless solid [yield 72%, m.p. 423–424 K; R_f = 0.67 (hexane–ethyl acetate, 4:1 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in ethyl acetate at room temperature.

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aryl and 0.98 Å for methyl H atoms. $U_{iso}(H) = 1.2U_{eq}(C)$ for aryl and $1.5U_{eq}(C)$ for methyl H atoms. The positions of methyl hydrogens were optimized rotationally.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 2012) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

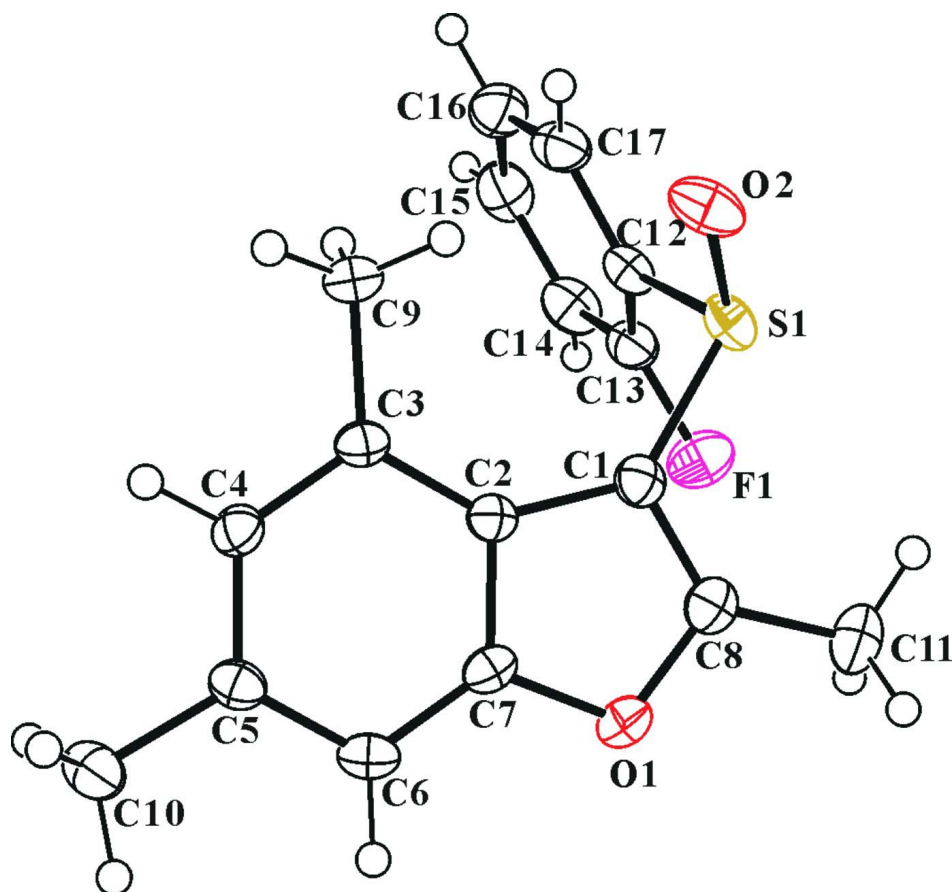
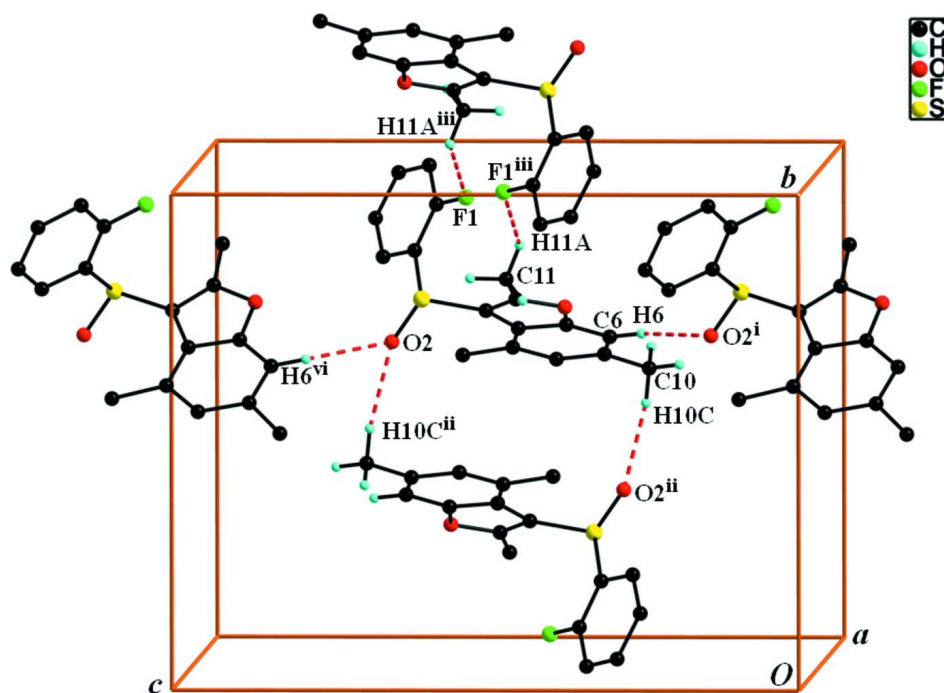
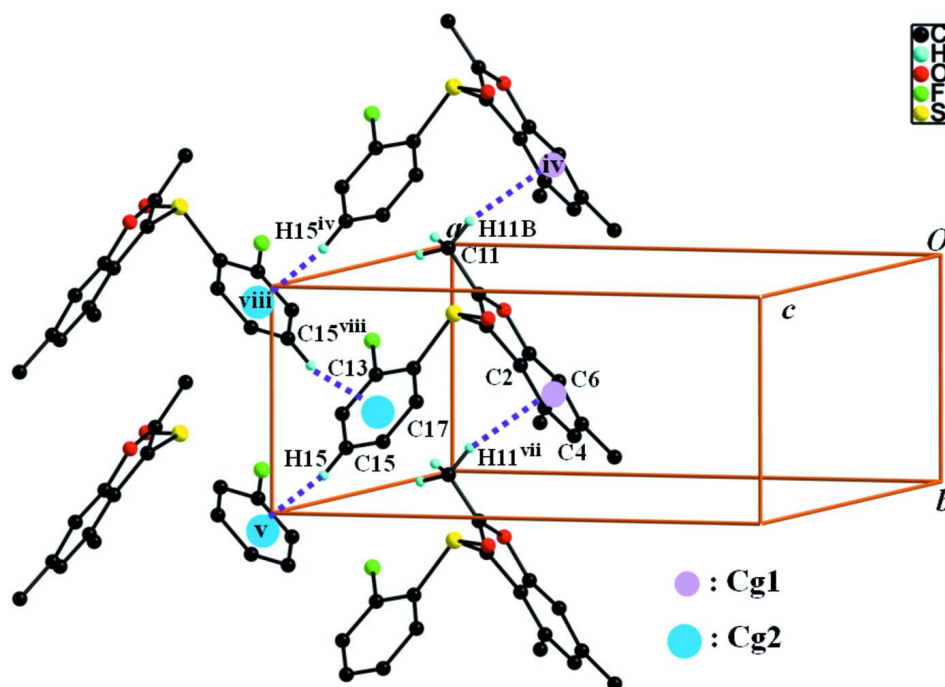


Figure 1

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius.

**Figure 2**

A view of the C—H \cdots F and C—H \cdots O interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (i) $x, -y + 1/2, z - 1/2$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x + 2, -y, -z + 1$; (vi) $x, -y + 1/2, z + 1/2$.]

**Figure 3**

A view of the C—H... π interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen-bonding were omitted for clarity. [Symmetry codes: (iv) $x, y - 1, z$; (v) $-x + 1, y + 1/2, -z + 3/2$; (vii) $x, y + 1, z$; (viii) $-x + 2, y - 1/2, -z + 3/2$.]

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Crystal data

$C_{17}H_{15}FO_2S$

$M_r = 302.35$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/bc$

$a = 13.6892\ (5)\ \text{\AA}$

$b = 6.0339\ (2)\ \text{\AA}$

$c = 17.1786\ (7)\ \text{\AA}$

$\beta = 92.741\ (2)^\circ$

$V = 1417.32\ (9)\ \text{\AA}^3$

$Z = 4$

$F(000) = 632$

$D_x = 1.417\ \text{Mg m}^{-3}$

Melting point = 423–424 K

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3545 reflections

$\theta = 2.4\text{--}27.5^\circ$

$\mu = 0.24\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Block, colourless

$0.32 \times 0.27 \times 0.12\ \text{mm}$

Data collection

Bruker SMART APEXII CCD
diffractometer

Radiation source: rotating anode

Graphite multilayer monochromator

Detector resolution: $10.0\ \text{pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)

$T_{\min} = 0.527$, $T_{\max} = 0.746$

13205 measured reflections

3277 independent reflections

2665 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 27.6^\circ$, $\theta_{\min} = 1.5^\circ$

$h = -17 \rightarrow 17$

$k = -7 \rightarrow 7$

$l = -22 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.114$
 $S = 1.07$

3277 reflections

193 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.6536P]$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.43 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.76961 (3)	0.18134 (7)	0.61409 (3)	0.02772 (14)
F1	0.95958 (8)	0.3216 (2)	0.55355 (7)	0.0387 (3)
O1	0.75116 (9)	0.2075 (2)	0.38753 (7)	0.0263 (3)
O2	0.68255 (10)	0.1904 (2)	0.66215 (8)	0.0380 (4)
C1	0.73907 (12)	0.2512 (3)	0.51663 (10)	0.0236 (4)
C2	0.68389 (12)	0.4305 (3)	0.47829 (9)	0.0205 (3)
C3	0.62608 (12)	0.6111 (3)	0.50021 (9)	0.0215 (3)
C4	0.58790 (12)	0.7440 (3)	0.44025 (10)	0.0235 (4)
H4	0.5496	0.8682	0.4538	0.028*
C5	0.60215 (12)	0.7073 (3)	0.36105 (10)	0.0236 (4)
C6	0.65664 (12)	0.5260 (3)	0.33935 (10)	0.0250 (4)
H6	0.6669	0.4938	0.2862	0.030*
C7	0.69534 (12)	0.3945 (3)	0.39892 (10)	0.0219 (3)
C8	0.77602 (13)	0.1244 (3)	0.45981 (11)	0.0256 (4)
C9	0.60497 (14)	0.6599 (3)	0.58346 (10)	0.0292 (4)
H9A	0.6580	0.7497	0.6073	0.044*
H9B	0.5999	0.5204	0.6122	0.044*
H9C	0.5432	0.7413	0.5852	0.044*
C10	0.55703 (14)	0.8621 (3)	0.30094 (11)	0.0313 (4)
H10A	0.5704	0.8081	0.2487	0.047*
H10B	0.5851	1.0105	0.3083	0.047*
H10C	0.4862	0.8687	0.3066	0.047*
C11	0.83800 (15)	−0.0766 (3)	0.46046 (13)	0.0355 (5)
H11A	0.9024	−0.0393	0.4415	0.053*
H11B	0.8070	−0.1895	0.4265	0.053*
H11C	0.8457	−0.1342	0.5137	0.053*

C12	0.84182 (13)	0.4201 (3)	0.64254 (10)	0.0249 (4)
C13	0.93069 (13)	0.4587 (3)	0.61046 (10)	0.0273 (4)
C14	0.99117 (14)	0.6298 (3)	0.63429 (11)	0.0344 (4)
H14	1.0519	0.6532	0.6110	0.041*
C15	0.96141 (16)	0.7673 (3)	0.69317 (12)	0.0375 (5)
H15	1.0016	0.8881	0.7102	0.045*
C16	0.87342 (16)	0.7297 (4)	0.72739 (11)	0.0368 (5)
H16	0.8536	0.8241	0.7680	0.044*
C17	0.81432 (14)	0.5551 (3)	0.70271 (10)	0.0313 (4)
H17	0.7547	0.5279	0.7271	0.038*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0300 (3)	0.0266 (2)	0.0265 (2)	−0.00358 (18)	0.00096 (18)	0.00813 (17)
F1	0.0313 (6)	0.0457 (7)	0.0401 (7)	0.0008 (5)	0.0103 (5)	−0.0076 (5)
O1	0.0266 (6)	0.0262 (6)	0.0264 (6)	0.0024 (5)	0.0039 (5)	−0.0054 (5)
O2	0.0360 (8)	0.0495 (9)	0.0292 (7)	−0.0118 (7)	0.0083 (6)	0.0102 (6)
C1	0.0242 (9)	0.0221 (8)	0.0244 (8)	−0.0027 (7)	0.0015 (7)	0.0010 (7)
C2	0.0209 (8)	0.0209 (8)	0.0199 (8)	−0.0026 (6)	0.0015 (6)	−0.0001 (6)
C3	0.0213 (8)	0.0242 (8)	0.0193 (8)	−0.0022 (7)	0.0037 (6)	−0.0018 (6)
C4	0.0223 (8)	0.0242 (8)	0.0245 (9)	0.0023 (7)	0.0041 (7)	−0.0009 (7)
C5	0.0217 (8)	0.0287 (9)	0.0205 (8)	−0.0022 (7)	0.0017 (7)	0.0016 (7)
C6	0.0241 (9)	0.0328 (9)	0.0183 (8)	−0.0036 (7)	0.0033 (7)	−0.0020 (7)
C7	0.0206 (8)	0.0227 (8)	0.0228 (8)	−0.0021 (7)	0.0047 (7)	−0.0048 (7)
C8	0.0232 (9)	0.0234 (8)	0.0302 (9)	−0.0032 (7)	0.0016 (7)	−0.0015 (7)
C9	0.0362 (10)	0.0315 (9)	0.0202 (8)	0.0035 (8)	0.0054 (7)	−0.0036 (7)
C10	0.0304 (10)	0.0378 (10)	0.0258 (9)	0.0015 (8)	0.0007 (8)	0.0055 (8)
C11	0.0341 (10)	0.0246 (9)	0.0483 (12)	0.0036 (8)	0.0062 (9)	−0.0013 (8)
C12	0.0245 (9)	0.0288 (9)	0.0213 (8)	−0.0003 (7)	−0.0012 (7)	0.0062 (7)
C13	0.0262 (9)	0.0321 (9)	0.0238 (8)	0.0027 (7)	0.0015 (7)	0.0033 (7)
C14	0.0280 (10)	0.0411 (11)	0.0338 (10)	−0.0068 (8)	0.0003 (8)	0.0078 (9)
C15	0.0418 (12)	0.0335 (10)	0.0361 (11)	−0.0062 (9)	−0.0083 (9)	0.0023 (8)
C16	0.0418 (12)	0.0397 (11)	0.0282 (10)	0.0054 (9)	−0.0051 (9)	−0.0039 (8)
C17	0.0292 (10)	0.0410 (11)	0.0237 (9)	0.0037 (8)	0.0010 (7)	0.0025 (8)

Geometric parameters (\AA , $^\circ$)

S1—O2	1.4830 (15)	C9—H9A	0.9800
S1—C1	1.7575 (18)	C9—H9B	0.9800
S1—C12	1.8014 (19)	C9—H9C	0.9800
F1—C13	1.354 (2)	C10—H10A	0.9800
O1—C8	1.367 (2)	C10—H10B	0.9800
O1—C7	1.382 (2)	C10—H10C	0.9800
C1—C8	1.357 (3)	C11—H11A	0.9800
C1—C2	1.458 (2)	C11—H11B	0.9800
C2—C7	1.397 (2)	C11—H11C	0.9800
C2—C3	1.409 (2)	C12—C13	1.379 (2)
C3—C4	1.388 (2)	C12—C17	1.382 (3)
C3—C9	1.502 (2)	C13—C14	1.373 (3)

C4—C5	1.401 (2)	C14—C15	1.385 (3)
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.385 (2)	C15—C16	1.384 (3)
C5—C10	1.503 (2)	C15—H15	0.9500
C6—C7	1.380 (2)	C16—C17	1.382 (3)
C6—H6	0.9500	C16—H16	0.9500
C8—C11	1.480 (2)	C17—H17	0.9500
O2—S1—C1	111.13 (8)	H9A—C9—H9C	109.5
O2—S1—C12	105.34 (9)	H9B—C9—H9C	109.5
C1—S1—C12	99.73 (8)	C5—C10—H10A	109.5
C8—O1—C7	106.64 (13)	C5—C10—H10B	109.5
C8—C1—C2	107.23 (15)	H10A—C10—H10B	109.5
C8—C1—S1	118.03 (14)	C5—C10—H10C	109.5
C2—C1—S1	134.65 (13)	H10A—C10—H10C	109.5
C7—C2—C3	118.16 (15)	H10B—C10—H10C	109.5
C7—C2—C1	104.22 (14)	C8—C11—H11A	109.5
C3—C2—C1	137.61 (15)	C8—C11—H11B	109.5
C4—C3—C2	116.43 (15)	H11A—C11—H11B	109.5
C4—C3—C9	120.71 (16)	C8—C11—H11C	109.5
C2—C3—C9	122.85 (15)	H11A—C11—H11C	109.5
C3—C4—C5	124.36 (16)	H11B—C11—H11C	109.5
C3—C4—H4	117.8	C13—C12—C17	118.43 (17)
C5—C4—H4	117.8	C13—C12—S1	120.65 (14)
C6—C5—C4	119.20 (16)	C17—C12—S1	120.58 (14)
C6—C5—C10	120.98 (16)	F1—C13—C14	118.80 (17)
C4—C5—C10	119.81 (16)	F1—C13—C12	118.61 (16)
C7—C6—C5	116.55 (15)	C14—C13—C12	122.59 (18)
C7—C6—H6	121.7	C13—C14—C15	118.30 (18)
C5—C6—H6	121.7	C13—C14—H14	120.9
C6—C7—O1	124.03 (15)	C15—C14—H14	120.9
C6—C7—C2	125.26 (16)	C16—C15—C14	120.28 (19)
O1—C7—C2	110.71 (15)	C16—C15—H15	119.9
C1—C8—O1	111.18 (15)	C14—C15—H15	119.9
C1—C8—C11	133.64 (18)	C17—C16—C15	120.22 (19)
O1—C8—C11	115.16 (16)	C17—C16—H16	119.9
C3—C9—H9A	109.5	C15—C16—H16	119.9
C3—C9—H9B	109.5	C12—C17—C16	120.14 (18)
H9A—C9—H9B	109.5	C12—C17—H17	119.9
C3—C9—H9C	109.5	C16—C17—H17	119.9
O2—S1—C1—C8	−135.67 (14)	C3—C2—C7—O1	−178.00 (14)
C12—S1—C1—C8	113.59 (15)	C1—C2—C7—O1	0.98 (18)
O2—S1—C1—C2	48.2 (2)	C2—C1—C8—O1	1.1 (2)
C12—S1—C1—C2	−62.50 (19)	S1—C1—C8—O1	−176.04 (11)
C8—C1—C2—C7	−1.21 (18)	C2—C1—C8—C11	179.30 (19)
S1—C1—C2—C7	175.18 (15)	S1—C1—C8—C11	2.2 (3)
C8—C1—C2—C3	177.45 (19)	C7—O1—C8—C1	−0.45 (19)
S1—C1—C2—C3	−6.2 (3)	C7—O1—C8—C11	−179.04 (15)

C7—C2—C3—C4	−2.3 (2)	O2—S1—C12—C13	177.90 (14)
C1—C2—C3—C4	179.19 (19)	C1—S1—C12—C13	−66.86 (15)
C7—C2—C3—C9	177.20 (16)	O2—S1—C12—C17	4.74 (17)
C1—C2—C3—C9	−1.3 (3)	C1—S1—C12—C17	119.98 (15)
C2—C3—C4—C5	1.0 (3)	C17—C12—C13—F1	177.51 (15)
C9—C3—C4—C5	−178.50 (17)	S1—C12—C13—F1	4.2 (2)
C3—C4—C5—C6	0.8 (3)	C17—C12—C13—C14	−2.1 (3)
C3—C4—C5—C10	179.86 (17)	S1—C12—C13—C14	−175.40 (14)
C4—C5—C6—C7	−1.3 (2)	F1—C13—C14—C15	−179.24 (17)
C10—C5—C6—C7	179.72 (16)	C12—C13—C14—C15	0.4 (3)
C5—C6—C7—O1	179.85 (15)	C13—C14—C15—C16	0.9 (3)
C5—C6—C7—C2	−0.1 (3)	C14—C15—C16—C17	−0.4 (3)
C8—O1—C7—C6	179.66 (16)	C13—C12—C17—C16	2.6 (3)
C8—O1—C7—C2	−0.38 (18)	S1—C12—C17—C16	175.89 (14)
C3—C2—C7—C6	2.0 (3)	C15—C16—C17—C12	−1.4 (3)
C1—C2—C7—C6	−179.06 (16)		

Hydrogen-bond geometry (Å, °)

Cg1 and Cg2 are the centroids of the C2—C7 benzene ring and the C12—C17 2-fluorophenyl ring, respectively.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C6—H6 \cdots O2 ⁱ	0.95	2.42	3.347 (2)	164
C10—H10C \cdots O2 ⁱⁱ	0.98	2.42	3.385 (2)	167
C11—H11A \cdots F1 ⁱⁱⁱ	0.98	2.54	3.160 (2)	121
C11—H11B \cdots Cg1 ^{iv}	0.98	2.69	3.476 (2)	138
C15—H15 \cdots Cg2 ^v	0.95	2.71	3.548 (2)	147

Symmetry codes: (i) $x, -y+1/2, z-1/2$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x+2, -y, -z+1$; (iv) $x, y-1, z$; (v) $-x+2, y+1/2, -z+3/2$.